

# 2C-B Synthesis

From 2,5-dimethoxybenzaldehyde.

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## List of Chemicals

- 100g 2,5-Dimethoxybenzaldehyde
- 10g Ammonium acetate
- 200g Nitromethane
- Isopropyl Alcohol (IPA)
- 500ml Tetrahydrofuran (THF)
- Anhydrous Calcium Chloride (eg, Drierite)
- NaOH
- HCl
- 20g Bromine
- Glacial acetic acid
- Methylene chloride
- 25 grams of LAH

## List of Materials

- 500ml boiling flask
  - 200ml boiling flask
  - condenser
  - electric heater/heating mantel
  - oil bath
  - boiling chips
  - filter paper
  - separation funnel
  - high-temperature distillation flask
  - 250ml filter flask
  - 2 250ml beakers
  - 500ml beaker
  - vacuum pump and hose
  - thermometer
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**DISCLAIMER#1:** It should be perfectly clear that 2C-B, also known under a variety of names such as "venus", "bromo", "erox", or "nexus", is currently a Schedule-I substance. In order to protect us from ourselves, our government has dutifully made it illegal to possess, manufacture, or distribute any amount of 2C-B for any purpose. Any persons caught breaking this law are subject to prosecution, imprisonment, dehumanization, demonization, humiliation, character defamation, intense personal harassment, and to top it all off - drug diversion. You've been warned.

**DISCLAIMER#2:** It should be made perfectly clear that laboratory chemistry is a very unforgiving art form. It can take years to master even the simplest of reactions and tiny mistakes can literally blow up in your face. Although this synthesis is not overly complex, it contains some hazards and will take a good deal of laboratory skill, some sophisticated equipment, and a couple of days to complete. Please use caution when testing the final product - it is always best to start off with minute amounts and build dosage slowly so as to minimize any unforeseen adverse effects.

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2C-B is not exactly easy to make but it is pretty straightforward. There aren't any very tricky reactions or especially messy procedures and the chemicals are not particularly suspicious to obtain (any more than anybody buying chemicals or lab equipment today is suspicious to the authorities). But, like any synthesis of this nature, patience and precision are most imperative. Be sure to make the measurements exact, keep temperatures precise and check the colors or consistency of all intermediates. A general chemistry background is also very helpful, but not necessary if you take the time to learn about what you are doing.

The basic precursor for synthesizing 2C-B is 2,5-dimethoxybenzaldehyde (2,5DMB). Although you can even make this compound, the procedure is fairly involved. For our purposes, it is best to start off purchasing this material.

The first procedure is to change the 2,5DMB into 2,5-dimethoxynitrostyrene. We do this by heating a mixture of 2,5DMB and nitromethane in the presence of a catalyst, ammonium acetate. Set up an

apparatus for refluxing. 'Refluxing' is basically boiling a solvent in such a way that the solvent continually evaporates, condenses, and is returned back to the boiling vessel. To do this we set up the apparatus as shown.

Place 100 Grams of 2,5DMB into a 500ml flask and add 200grams of nitromethane. Most of the solids should dissolve but if they don't immediately don't worry - they will once you start heating them. When most of them are dissolved, add 10 grams of ammonium acetate. Place the flask into an oil bath on an electric hotplate, connect the condenser and start a slow water flow through the condenser. DO NOT use open flame in any of these procedures. Nitromethane is very flammable! It is, in fact, rocket fuel. It's dangerous enough using an electric hot plate because of the sparks produced when the thermostat clicks on and off, but if you keep reasonable ventilation and a watchful eye on things, you should be okay. Turn the stove up to about 100 degrees celsius, the boiling point of nitromethane, and adjust it until the solution is boiling nicely. In a short time the color of the solution will turn yellow. Four hours later, when you are done refluxing, the color should be a deep red.

Immediately set up for vacuum evaporation. If you let the solution cool off too much it may spontaneously crystalize, making it more difficult to purify, so do a little planning ahead of time. Turn the heat off, detach the condenser from the flask and add a few boiling chips to mixture. Boiling chips provide a very large surface area for lots of small boiling bubbles to form. This will keep the solution boiling smoothly without any "bumps" or big erratic bubbles. Connect the vacuum hose and apply a vacuum very gently at first and build up to a rolling boil without boiling up into the hose. As the solution evaporates, the flask will cool down and you will have to adjust the heat of the hot plate to keep a steady boil going. Eventually, you will remove enough solvent and the whole mass will crystalize into bright pumpkin-orange crystals.

Turn off the heat and remove the vacuum hose from the flask. Always remove the hose from the flask before turning off the water. Otherwise, as the pressure drops water may be sucked into your product. Add 100ml of boiling isopropyl alcohol (IPA) to the mass and stir it as well as you can, breaking up all of the clumps. Your hot plate may still be hot and if you are working near it be sure NOT to splash any of this nitrostyrene onto the hot surface. The resulting fumes are a very potent tear gas, something you don't want to experience! Let the flask cool and then pour off the red-colored alcohol solution. Add another 100ml of boiling IPA to the crystals and rinse as before. Rinse at least two more times or until needle-like crystals form out of the amorphous orange mush. Filter these crystals off and air dry completely. There must not be any alcohol left in the crystals, as this will ruin the lithium aluminum hydride (LAH) in the next step. This is 2,5-dimethoxynitrostyrene (2,5DMNS)

Dissolve 50 grams of 2,5DMNS in 300ml of anhydrous (dry) THF. If you cannot find anhydrous THF, then you will have to dry it the best you can. Do this by adding a drying agent (such as Drierite, or anhydrous Epson salts) to the THF, let it sit overnight or longer, and then filter it out. This should get most of the moisture out of the THF that would otherwise ruin your expensive LAH.

Arrange your refluxing setup with a 2-liter, 2-neck flask in the oil bath, a magnetic stir bar in the flask and a drying tube attached to the condenser. This can be a spare tube, plugged with cotton and filled with Drierite, that is attached to the open end of the condenser to prevent moisture from the air entering the reaction. Put 750ml of dry THF into the two neck flask and start to stir at a slow speed. SLOWLY add 25 grams of LAH to the stirring THF (LAH is nasty, dangerous stuff. It ignites in the presence of water, it's poisonous to breathe, to touch etc. Read up on it...). If you start adding the LAH too fast it may spark, so take your time. It fizzes as it hits the THF, so add it carefully and let it easily mix into the THF. Now you are going to add your nitrostyrene solution. This is the reason for the two neck flask. As you add this to the swirling THF, it fizzes up pretty vigorously. Heat is generated and the THF starts to evaporate. Get the water running through the condenser and carefully add a little of the nitrostyrene solution (2,5DMNS) through the extra neck. When the fizzing starts to catch up with you, stopper the neck up quickly to keep the fumes from escaping and let the reaction calm down and cool down a little. Repeat this many times until you have added all of the nitrostyrene. When you are done you should have a dirty gray solution fizzing away nicely. Slowly raise the temperature until reflux is reached and hold it there for 24 hours.

Turn off the heat but leave the stirrer on and let the flask cool down. Once cold, SLOWLY add isopropyl alcohol (IPA) dropwise to neutralize any excess LAH. This will cause considerable fizzing. Make sure you are stirring while you add the IPA; if not, a layer of IPA will form and then any stirring that is done will result in the whole solution jumping right out of the flask. Take your time with this until there is no reaction with the addition of any more IPA.

This gray aluminum slush is nearly impossible to filter out of the solution, but with the addition of 50ml of 15% NaOH it becomes a white solid, and quite filterable. Now filter out the solids (you will be keeping the THF solution), and then wash the filter cake with a little extra THF. Combine the THF filtrate and the washings into a boiling flask.

Now you must remove the THF from the mixture. This is done by simple distillation under vacuum. Set up the apparatus as shown. Add a few boiling chips to the flask and place it in the oil bath. Start the vacuum and increase until full strength is reached. As the THF evaporates, the flask cools down and needs more pressure to cause boiling. Finally, use the hot plate to cause steady boiling. Eventually you will be left with a dark-brown/goldenish oily residue. This is very crude 2,5-dimethoxyphenethylamine, which you need to purify.

Suspend the oily residue in 1 liter of distilled water and make it slightly acidic (pH of around 4) by adding HCl. The 2,5DMPEA will become a water-soluble hydrochloride salt and dissolve into the water; other reaction byproducts will remain water-insoluble and can be easily removed. Add the solution to a suitably sized separation funnel and add 50ml of methylene chloride. Shake vigorously and let the phases separate. Drain out the methylene chloride. Repeat this washing with three more 50ml portions of methylene chloride. This should remove much of the solution's color and it will remain only slightly yellow. We must now freebase the solution before our final purification by fractional distillation.

Make the solution very basic by slowly adding a strong solution of NaOH. The mixture will immediately take on a milky appearance. Shake the mixture vigorously for a few minutes to make sure the 2,5DMPEA HCl is converted to the freebase. This freebase is a dark oil which will separate and start to settle out. Extract the oil by washing, as above, with portions of methylene chloride, but this time keep the washings. Combine all the methylene chloride washings and remove the solvent by distillation as above for removing THF. You will be left with around 50ml of a dark oil.

Now we will distill the oil under reduced pressure and at a relatively high temperature. To do this we use a slightly different setup than the one used to remove the solvents. We won't need the condenser in this case and the flask should be much smaller (around 100 to 250ml) to adequately accommodate the smaller volume of oil. Setup the apparatus as shown. Make sure you use a water trap. Any cold water entering the system will crack the flasks and ruin the procedure. Also make sure to add boiling chips to the flask. Oils tend to really spatter and the chips are absolutely necessary. Bring the system up to full pressure slowly. There will probably still be some methylene chloride that will boil off and end up on the receiving vessel. This is ok. As we heat the system it will eventually evaporate out of the flask. At full water pressure, the oil bath will need to reach 195 Celcius before the 2,5DMPEA will start to come over as clear, waterlike oil. Keep the temperature and pressure constant as the oil slowly drips over. This will take some time depending on your exact setup. When you are satisfied that you have distilled out all the product turn off the heat and disconnect the vacuum from the flask. You should have around 20 grams of pure water-white 2,5DMPEA.

Now we will brominate the 4 position of the benzene ring of our 2,5DMPEA. We do so with pure elemental bromine. Bromine is by far the nastiest stuff you'll ever want to come across. As soon as you open the bottle it will start to crawl out. If you noticed how well packed your bottle of bromine was, you were probably expecting something like this. Use this only in a fume hood if possible. Don't even open the bottle in your house or the whole house will smell like bromine for days and days. Fortunately, we don't have to deal with it for very long. If you have no access to a fume hood (which is probably the case) then at least pick a well ventilated area, away from any nosey public, and have a large jar with an airtight seal available. Place your 2,5DMPEA in a 250ml beaker and weigh it on a

digital scale. Add to it this same amount of glacial acetic acid. Place another 250ml beaker on the scale, quickly open the bromine and weigh out the same weight as your 2,5DMPEA. Add to it that same amount of glacial acetic acid and quickly add the 2,5DMPEA solution to it. After a few quick stirs, seal it in the large airtight jar. The beaker will heat up but it shouldn't be a problem with these quantities. If you scale this up you may need to rig up an external cooling water bath. Light yellow solids will slowly form and in a few hours the solution will cool down. The beaker should pretty much now be solid with light yellow crystals of 2C-B-HBr.

This 2C-B-HBr has many associated forms involved with it and for a consistent, predictable drug we will need to convert it into the hydrochloride salt. Take the whole mass of 2C-B-HBr and filter it. Rinse a few times with cold glacial acetic acid. Place the still-wet crystals into a 500ml beaker and slowly add a 40% NaOH solution. Stir briskly and a dark oil will start to settle out. Separate this oil, as you did above, by extracting with methylene chloride. You will also distill this oil, under pressure and at high temperature, as you did above, and again collect a clear oil. This is pure 2C-B freebase.

The final step is making the hydrochloride salt of 2C-B. This is done very easily. Since 2C-B is not soluble in water, we don't have to worry about completely anhydrous solvents that are usually the case when crystallizing the final products. Add the freebase oil to 100ml of distilled water and slowly add acetic acid while stirring until the freebase dissolves. This won't take very much acetic acid. Get this solution stirring with the magnetic stirrer and slowly add concentrated HCl. A white precipitate of 2C-B-HCl will immediately form. Continue to add the HCl until the solution is thick with fine white crystals. Stop the stirring, and when the crystals settle out, decant the water into another beaker. Add a little water to the crystals, stir and let settle. Add this wash to the beaker with the original decanted solution, put it back on the stirrer and add more HCl. More crystals should form. Repeat this process until you are satisfied that all the crystals have formed. Now add all the crystals together - add water, stir, let settle and decant repeatedly until the wash water has a fairly neutral pH. Now you can filter the crystals and air dry. This is fairly pure 2C-B-HCl. Enjoy.

Dosage: in PIHKAL, Shulgin recommends 12-24mg, which is a good range to work with when taken orally. An alternate means of administration is by sniffing. I have found this much more effective. Cut the dosage in half and expect to feel results within 10 minutes or less! **Note:** Sniffing can be very painful! It's best to do smaller amounts than the whole dose all at once. As known from above, 2C-B is not water soluble and will sit in your nose burning for while - and it does burn. But the pain goes away in short time and the results are worth it. Experiment!

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## Additional commentary

Apparatus wise the real problem is with the distillation of the 2CH and 2C-B free base. Basically the setup described there will cause the flask contents to bump really badly and boil over straight into the receiver, there may be chemists who could get that setup to work but I don't think I could. It is much much better to use a 2 neck adapter with the still head in one and in the other a capillary airleak tube made by stretching out 8mm OD borosilicate cap. tubing in a flame until it narrows then breaking it in the middle of the narrow bit. With a steady stream of bubbles being sucked through the liquid the bumping is much reduced. The extra distance also means any boil over can be stopped before it messes up your hard work.

The removal of the nitromethane and dichloromethane using the apparatus on the lycaeum is fine but it must be pointed out that adding antibump granules to a hot/almost boiling liquid is a very bad idea....

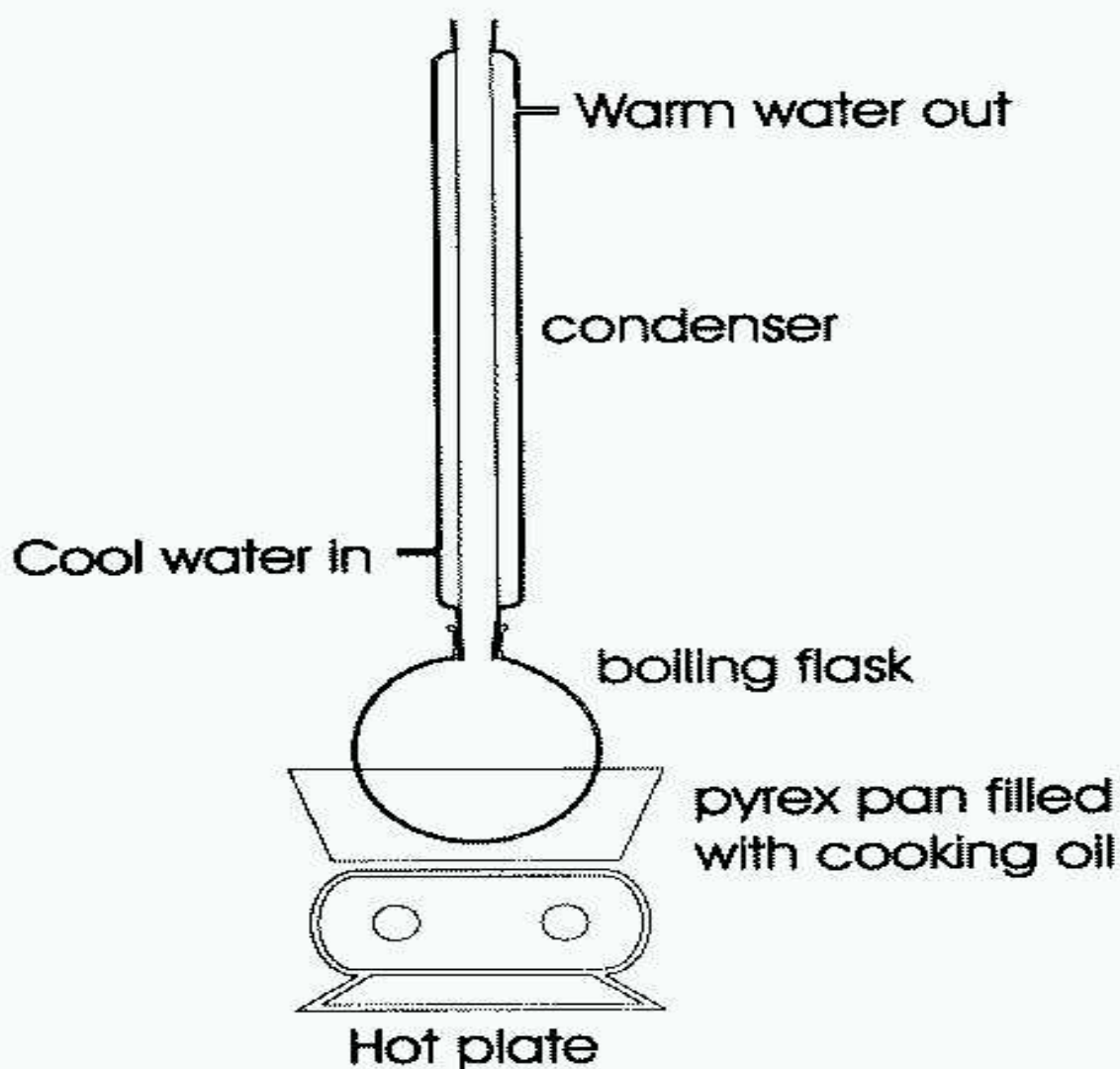
There are a few other apparatus points but these are fairly trivial.

Chemical wise, it seems very important for decent yield that the ammonium acetate is dry, the

commercial stuff looks like clear sugar and contains a fair amount of water, standing over calcium chloride in a dessicator-or an ice cream tub at a push dries it sufficiently. The THF/nitrostyrene solution should really be added using a pressure equalized dropping funnel over 4-5 hours, If your adding it by hand there is a serious risk of adding to muchat once either by accident or because you're bored and want to get on with it 4 hrs is a very long time to be doing nothing except adding tiny drops of solution :)

Maybe I'm being picky here and I suppose a half decent chemist would use some common sense. But the information can't hurt.

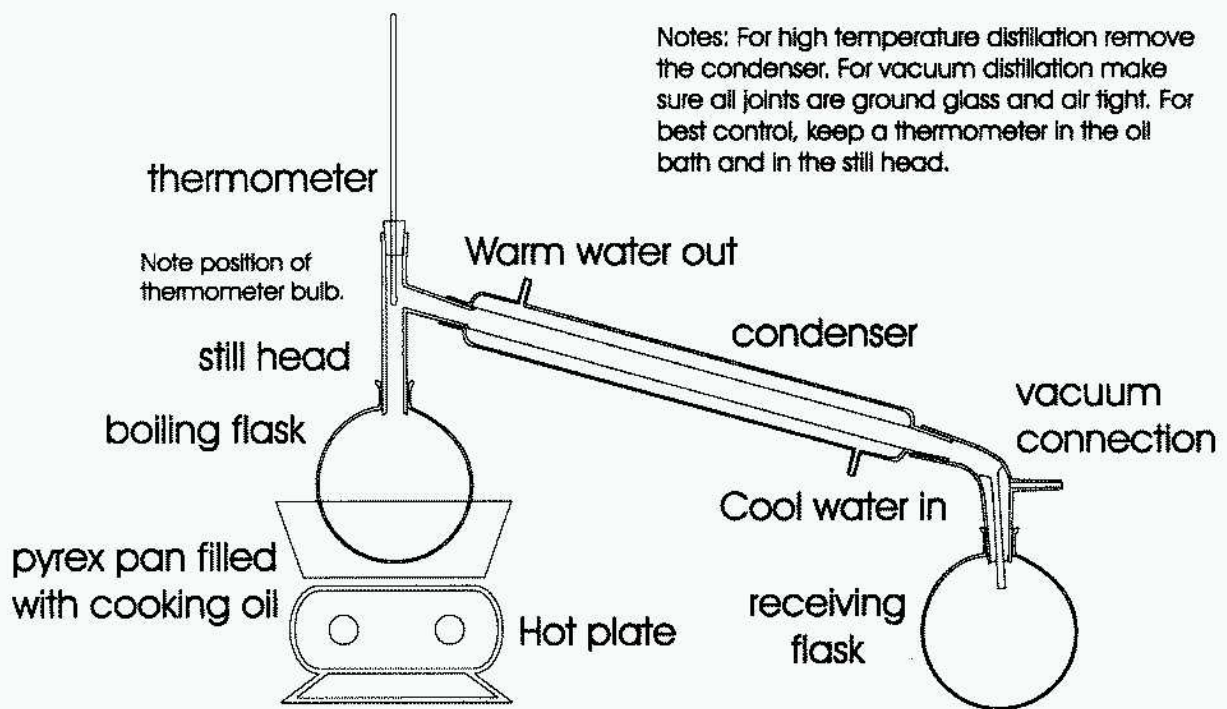
Diagram of reaction vessel with reflux condenser



## Basic reflux set-up

Everything is secured with clamps and ring stands

Diagram of vacuum distillation apparatus



## Basic distillation set-up

Everything is secured with clamps and ring stands

Reaction Scheme: 2,5-dimethoxybenzaldehyde to 2C-B

## The Chemical Process

